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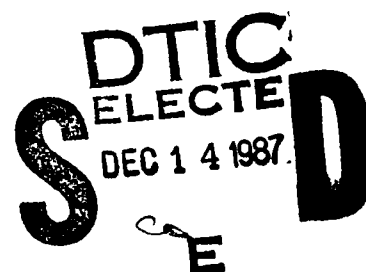
POLYMERIC MATERIALS FOR ELECTRO-OPTIC TESTING

University of California, Davis

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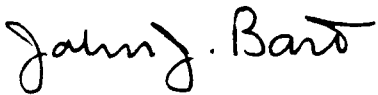
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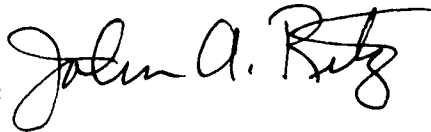| 2a SECURITY CLASSIFICATION AUTHORITY N/A	3 DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited
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19 ABSTRACT (Continue on reverse if necessary and identify by block number)

This report investigates the possible use of Langmuir/Blodgett films to electro-optically test integrated circuits. There is a brief description of what Langmuir/Blodgett films are, how they are grown and deposited on a material, and the electro-optic effects in Langmuir/ Blodgett films. Stephen Kowel has experimented with several different types of organic dyes mixed in the films to increase the electro-optic effect in the films. The bulk of his research has been in perfecting his deposition technique as well as increasing the electro-optic effect in the film. Also, he has done in-depth work developing a technique to preferentially align the dipoles in the film in order to allow for use of stronger electrical fields. Although Langmuir/Blodgett films were not actually deposited on silicon integrated circuits, there is great promise Langmuir/Blodgett films can be used to electro-optically test integrated circuits.

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EVALUATION

This effort's major objective was to investigate the possible use of organic films to produce an electro-optic medium which could be deposited on an IC (Integrated Circuit). Development of such a film would enable an engineer to optically test the internal nodes of an IC. Thus, test engineers would have a powerful technique to aid the debug of an IC or to more fully characterize the device.

The majority of the work performed by Dr. Kowel and his associates centered on the development and perfection of the deposition technique of the Langmuir/Blodgett film. Langmuir/Blodgett films must be deposited in monolayers. In order for them to exhibit a large electro-optic co-efficient the molecules in the monolayer must be in the proper alignment. Typically, 100 monolayers must be deposited to show a significant electro-optic coefficient with each layer properly aligned. In order to accomplish this task, Dr. Kowel used electric fields on each monolayer to get the proper alignment. After several iterations, they developed a reliable technique to deposit quality films. Once a reproducible deposition technique had been perfected, Dr. Kowel began to test depositing the film on a silicon test structure. This test structure is an interdigitated capacitance sensor. When a voltage is applied to the structure an electric field will be generated. When a linearly polarized light beam passes through the film over the test structure, there should be a change in the polarization as a function of applied voltage. Using this test scenario, Dr. Kowel proved one could optically make voltage measurements with a Langmiur/Blodgett film deposited on a silicon test device.

The next step in Dr. Kowel's research is to deposit the Langmuir/Blodgett film on an actual functioning silicon integrated circuit and make optical measurements of voltage of internal nodes of the device. I believe the use of organic films as electro-optic mediums holds great promise and will be a viable technique to do non-contact testing of internal nodes of integrated circuits.

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1.0 Introduction

The purpose of this effort was to investigate the use of Langmuir/ Blodgett films for the electro-optic testing of integrated circuits. Based on the use of materials such as $LiNbO_3$ for the testing of ultra-fast discrete devices, it appears that the development of similar materials which could be placed with good topographic coverage on an integrated circuit would allow for the optical testing of internal nodes. Such a material must exhibit linear electro-optic effects which are several orders of magnitude greater than materials such as $LiNbO_3$. Based on this requirement the electro-optic effects in the organic materials currently under investigation in our laboratory were evaluated.

1.1 Background

The science of monolayer surfaces and assembled monolayers is relatively young, being pioneered in the 1880's by Agnes Pockels. In the 1930's Katherine Blodgett and Irving Langmuir devised ways of both studying and depositing monolayers and developed what is commonly referred to as the Langmuir/Blodgett (L/B) technique. In view of this, much of the work being done today is basic research which will help to explain the fundamental structure and growth of L/B films. The work being done at the University of California at Davis addresses not only these issues but considers some of the important applications these materials will have, primarily in the area of nonlinear optics.

Under this contract our laboratory has been tasked with investigating the use of L/B films for the testing of integrated circuits. This application involves the growth of L/B films on integrated circuits and the subsequent use of the optical properties of the film to read voltages from the surface of the device. If sufficient voltage sensitivity is found, it should be possible to read voltages from the internal nodes of the integrated circuit with a laser probe. It will then be possible to develop a probing technique which is analogous to electron-beam testing, but does not require the use of a potentially damaging electron-beam or the inconvenience of a vacuum system.

By using broad illumination on the device, it should also be possible to generate an image containing voltage information. This offers the possibility of developing a optical analogue to dynamic fault imaging, whereby a device under test could be compared with a known good device or with stored images.

1.2 Deposition of Langmuir/Blodgett Films

The growth of monolayer or assembled monolayer films is dependent on the transfer of a monomolecular layer from the liquid surface to the substrate. In the L/B technique this is accomplished by creating a monolayer of material on a subphase liquid, which is highly purified water or a salt solution, and then dipping the substrate through the monolayer. Depending on the preparation of the substrate (hydrophilic or hydrophobic) the first monolayer may be deposited on the downstroke (for hydrophilic substrates) or only on the upstroke (for hydrophobic substrates). Because the monolayers can be deposited in a somewhat controllable manner the L/B technique offers the advantage that thin layers with anisotropic properties can be produced. The anisotropy of the films is the source of optical properties (such as the electro-optic effect) which have a number of interesting applications. Several review articles explain both the growth of L/B films and discuss their applications [1,2].

Currently in use at our lab is a Joyce Loebl Model 4 Langmuir Deposition Trough, and a Joyce Loebl Langmuir Minitrough installed in a class 100 modular clean room. Using this system, studies have been made of deposition of both standard L/B materials, such as fatty acids, and materials containing optically active merocyanine and hemicyanine dyes. Mixtures of the dye with robust polymers such as PMMA have been deposited successfully on glass slide substrates. The growth of oriented layers of hemicyanine dye on both glass and Si wafers has been demonstrated. This dye seems to show the most promise because of its optical properties and its stability. Each monolayer of the dye is on the order of 25 Angstroms thick, and up to 100 Z type layers (deposition on the upstroke only) have been

produced. This indicates a total film thickness of approximately 2500 Angstroms.

We have developed a real-time sensor of interdigitated thin film metal fingers to monitor the film growth during deposition. The sensor is a capacitance device which is sensitive enough to note the deposition of one monolayer of material. This device is useful not only for basic research to understand the film growth, but also demonstrates the ability to deposit a L/B film on a substrate with metallized pattern, a topography which will be encountered when the films are deposited on integrated circuits.

1.3 Measurement of Second Harmonic Generation

Second harmonic generation (SHG), also known as frequency doubling, occurs in materials where a nonlinear mechanism is both present and can couple to an electromagnetic wave in the optical region. SHG is of consequence in this work as its presence indicates that a linear electro-optic effect should exist, as the linear electro-optic effect is the mixing of a low frequency electronic signal with the optical beam, instead of the mixing of the beam with itself, as is found in SHG.

SHG was observed in our laboratory on films of hemicyanine which were 1-20 layers (25-500 Angstroms) thick. In these experiments, a Nd:YAG laser was used to generate a primary beam at 1064nm which was sent through the film sample. Filters were then used to remove the 1064nm light and a photodetector was used to detect the presence of the second harmonic signal at 532nm. The results of these experiments were published [3], and further experiments are being carried out to determine the exact components of the second harmonic tensor. Determination of the exact components of the tensor will be of great value in understanding the structure of the films and in determining which orientations of the film will produce the greatest linear electro-optic effect.

2.0 Accomplishments to Date

During the period of this contract, the deposition of hemicyanine dye on several glass substrates, and with various subphase (the liquid on which the monolayer is spread) solutions were examined. Deposition on silicon was briefly examined and found to be similar to deposition on glass. It was discovered that the subphase solution affects the deposition of the material and can actually aid or prevent deposition on either the upstroke or downstroke. An even greater effect is seen on the optical properties (SHG coefficients) of the material. The deposition ratios used as diagnostics during film deposition do not seem to be a good indicator of the relative optical merit of the film. Films which have only indicated deposition on the upstroke and would seem to be completely oriented do not necessarily have large SHG coefficients. However, it has been discovered that an ammonia subphase allows for production of films with high SHG coefficients which increase in a greater than linear fashion with the number of layers deposited. This indicates that the films posses a relatively high degree of order and show great promise for materials for SHG.

An experiment was performed to determine if the linear electro-optic (LEO) effect was observable in the hemicyanine dye but no effect was seen in a 100 layer sample. A detailed study of the electro-optic coefficients in other organic and inorganic materials was made and based on these results it was seen that if the electro-optic coefficients in the hemicyanine dye are an order of magnitude greater than those observed in $LiNbO_3$ the change in intensity will be on the order of one part in 10^6 for films of 100 layers, and electric fields on the order of 10^6 V/m. The experimental setup used is illustrated in Figures 1 and 2. It was determined that this experiment was not sensitive enough to detect the small changes in polarization induced by the applied field.

Several experiments have been performed using electric fields on the monolayer both while on the subphase and during deposition, in an effort to increase the ordering of the dipoles. Using vertical electric fields while the monolayer is on the subphase, small changes in

surface pressure have been observed, indicating that additional ordering of the monolayer is taking place.

Some preliminary work was performed to determine what the topographic coverage of the films was. SEM examination was used but due to the thinness of the films and the necessity of coating the sample with a thin conducting layer, (gold was used in this case) it was not possible to detect any defects in the films. Examination using an optical microscope has revealed some clustering of dye in the dye/PMMA mixtures but does not indicate such behavior in the pure dye films. Because of the relative thinness of the films fluorescence experiments could not be performed. Deposition of a high number of layers will be necessary to determine topographic coverage properties.
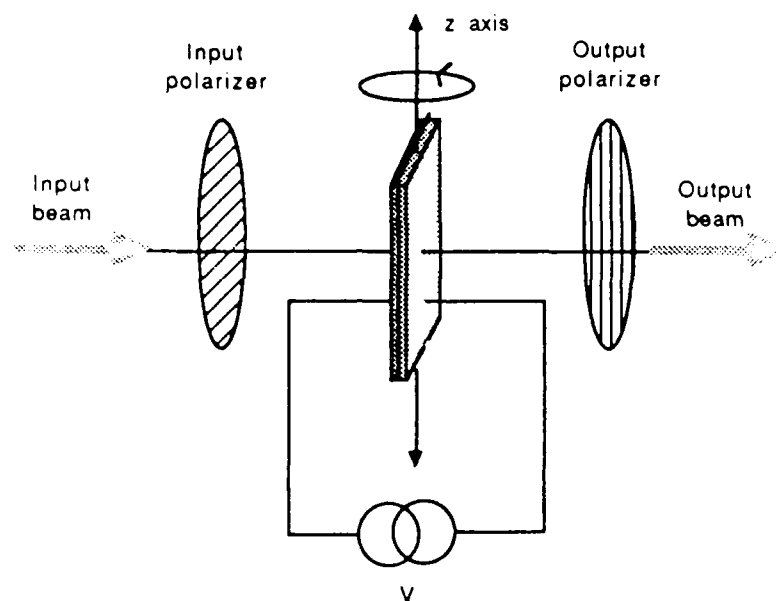
Figure 1a: Measurement of the longitudinal electro-optic effect when the applied electric field is perpendicular to the deposited L/B film. By rotating the sample slightly about the z axis, the transverse component can also be measured.
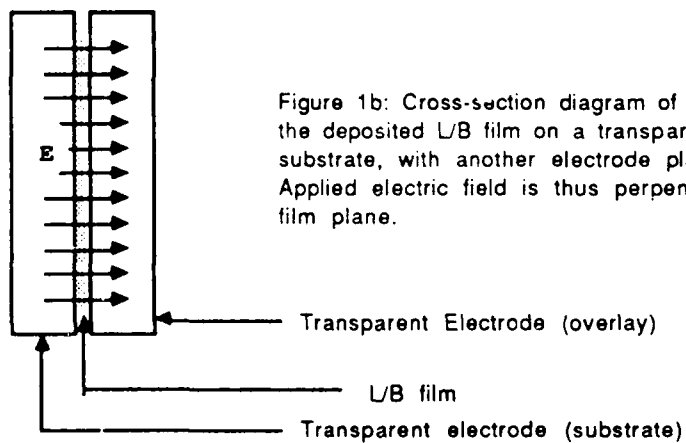

Figure 1b: Cross-section diagram of sample, showing the deposited L/B film on a transparent electrode substrate, with another electrode placed on top. Applied electric field is thus perpendicular to the film plane.
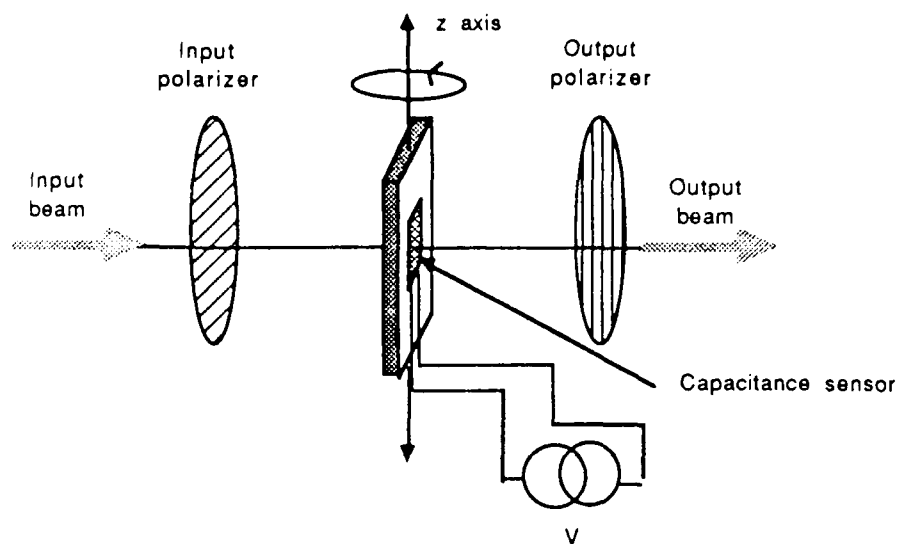
Figure 2a: Measurement of the transverse electro-optic effect when the electric field due to the capacitance sensor is parallel to the deposited film. In this case, a slight rotation of the sample about the z axis allows for measurement of the longitudinal component of the electo-optic effect.
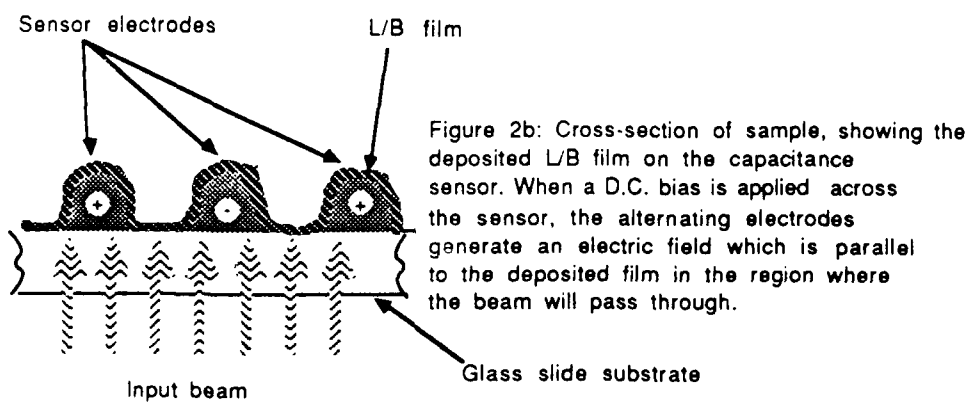

Figure 2b: Cross-section of sample, showing the deposited L/B film on the capacitance sensor. When a D.C. bias is applied across the sensor, the alternating electrodes generate an electric field which is parallel to the deposited film in the region where the beam will pass through.

3.0 Electro-Optic Effects in Langmuir Blodgett Films

An incident optical beam of linearly polarized light traveling along the z axis can be considered to be an electromagnetic wave $E(\omega,t)$ which can be resolved into two components on the x and y axes:

$$E_x(\omega,t) = E_x^{(0)} \exp[-k_x\omega z/c] \cos[\omega t - (n_x\omega z)/c]$$

$$E_y(\omega,t) = E_y^{(0)} \exp[-k_y\omega z/c] \cos[\omega t - (n_y\omega z)/c] \qquad (3-1)$$

Propagation of the optical beam through the material is completely described by the refractive indices n_x and n_y, and the absorption coefficients k_x and k_y.

If a propagating electromagnetic wave is resolved into components as in equation 3-1, polarization of wave is described by the amplitudes. $E_x^{(0)}$ and $E_y^{(0)}$, and any phase difference θ between the two components. For example, light which is linearly polarized at 45° to the x and y axes has amplitudes $E_x^{(0)} = E_y^{(0)} = E^{(0)}/\sqrt{2}$ and a phase angle which is equal to zero. If the wave propagates through a material which has equal complex indices of refraction along both axes, the transmitted portion of the beam will retain its initial polarization. If however, the indices of refraction along the axes are different, the polarization of the transmitted wave will differ from that of the incident wave. In particular, if the real portions of the indices differ, a phase delay Γ will be introduced such that;

$$\Gamma = (n_x - n_y)2\pi d/\lambda_0 \qquad (3-2)$$

where d is the thickness of the material and λ_0 is the wavelength of the wave in vacuum. The transmitted wave will no longer be linearly polarized but will instead have an electric field vector which traces out an ellipse when followed over one wavelength distance in space. A material producing such a change in polarization is said to be birefringent.

Changes in polarization can be converted to changes in amplitude through the use of polarizing filters. If a polarizing filter is placed at 90° with respect to the input polarization, only the birefringence induced portion of the light will be transmitted, with the resulting output intensity being equal to [4]

$$I_0 = I_i \sin^2(\Gamma/2) \qquad (3\text{-}3)$$

If the optical input signal is elliptically polarized to begin with, the modulator is considered to be optically biased and when biased at the 50 percent transmission point, small changes in phase will be seen as changes in intensity according to

$$I_0 = I_i (1/2 + \Gamma/2). \qquad (3\text{-}4)$$

In order to determine what phase changes an external field can induce, it is necessary to describe the indices of refraction as a function of applied electric field. Note that in the absence of any perturbing field the indices of refraction are described by the symmetric impermeability tensor η_{ij}. Upon suitable rotation the tensor becomes diagonal and the indices of refraction n_x, n_y, n_z, and are equal to η_{ii}, η_{jj} , and η_{kk}. It is also useful to note that a relationship between the indices of refraction can be derived [5] which is the equation of a general ellipsoid with major axes parallel to the x, y and z directions. The equation is

$$(x/n_x)^2 + (y/n_y)^2 + j(z/n_z)^2 = 1 \qquad (3\text{-}5)$$

and the ellipsoid is known as the index ellipsoid or the optical indicatrix.

In a material having electric birefringent properties, an applied electric field will induce changes in the impermeability tensor which can be described in a series of terms;

$$\eta_{ijk}(E) - \eta_{ijk}(0) = \Delta\eta_{ijk} = r_{ijk} E + s_{ijk} (E \cdot E)+... \qquad (3\text{-}6)$$

Where the tensor r contains what are referred to as the linear (or Pockels) electro-optic coefficients, and the tensor s contains the quadratic (or Kerr) electro-optic coefficients.

The r tensor initially has 27 components, but will be a symmetric tensor, just as the dielectric tensor ε is. This allows for the permutation of the indices i and j and the reduction of the number of components from 27 to 18. The standard notation for the contraction is

$$r_{1k} = r_{11k}$$
$$r_{2k} = r_{22k}$$
$$r_{3k} = r_{33k}$$
$$r_{4k} = r_{23k} = r_{32k}$$
$$r_{5k} = r_{13k} = r_{31k}$$
$$r_{6k} = r_{12k} = r_{21k}$$
$$k = 1,2,3.$$

The change in the impermeability tensor is then given by

$$\Delta\eta_i = \begin{bmatrix} r_{11} & r_{12} & r_{13} \\ r_{21} & r_{22} & r_{23} \\ r_{31} & r_{32} & r_{33} \\ r_{41} & r_{42} & r_{43} \\ r_{51} & r_{52} & r_{53} \\ r_{61} & r_{62} & r_{63} \end{bmatrix} \begin{bmatrix} E_x \\ E_y \\ E_z \end{bmatrix} \qquad (3\text{-}8)$$

The change in the index ellipsoid due to the applied electric field is given by

$$(x/n_x)^2 + (y/n_y)^2 + (z/n_z)^2 + \Delta\eta_i = 1. \qquad (3\text{-}9)$$

The resulting equation will often times contain cross terms which do not allow for a separation of the changes in the individual indices of refraction. Suitable rotation of the coordinate system eliminates the cross terms and allows for a description of the individual changes in the indices as a function of the applied field [6]. The changes in the individual indices of refraction result in a phase delay Γ given by equation (3-2). The resulting change in polarization can be converted to a change in intensity by the use of suitable polarizing filters.

In hemicyanine, the second harmonic generation coefficients indicate that the largest component in the electro-optic tensor is the r_{33} component. This component corresponds to the change in the index of refraction in the direction of the dipole moment of the dye molecules, with the applied field parallel to the dipole moment. In the hemicyanine dye, the dipole moment is perpendicular to the surface and thus the largest electro-optic is expected due to the component of the electric field which is perpendicular to the surface. If an integrated circuit were to be covered with a multilayer L/B film, the electric field component which would modulate the indices of refraction would be the vertical fringing fields. For a typical integrated circuit these fields are on the order of 10^2 - 10^3 V/m. The resulting phase delay is given by

$$\Gamma = (2\pi \ L)/\lambda[(n^3/2)r_{33}E_z] \qquad (3\text{-}10)$$

where L is the effective path length in the material and λ is the wavelength in vacuum. Calculations of the phase delay as a function of r were made and are illustrated in Figure 3a. The field was taken to be 10^3 V/m, $\lambda = 6328$ μm, and n, the index of refraction for hemicyanine has been determined to be 2.65. The results of these calculations show that for thin L/B films to be employable for testing purposes, the r coefficient will have to be on the order of 500 x 10^{-12} m/V, as compared to the largest r for $LiNbO_3$ which is 28 x 10^{-12} m/V. While it is possible that the films of hemicyanine will posses an r coefficient of this magnitude, it will be necessary to produce 50-100 layer films which show good SHG properties before the r coefficients can be measured.

Another possibility is to develop deposition techniques in which the dipole moment is oriented in one direction parallel to the surface on the integrated circuit. This orientation would allow for the employment of the horizontal fields between conductors, which are on the order of 10^6 V/m. Calculations of the phase delay as a function of r for this field are shown in Figure 3b.
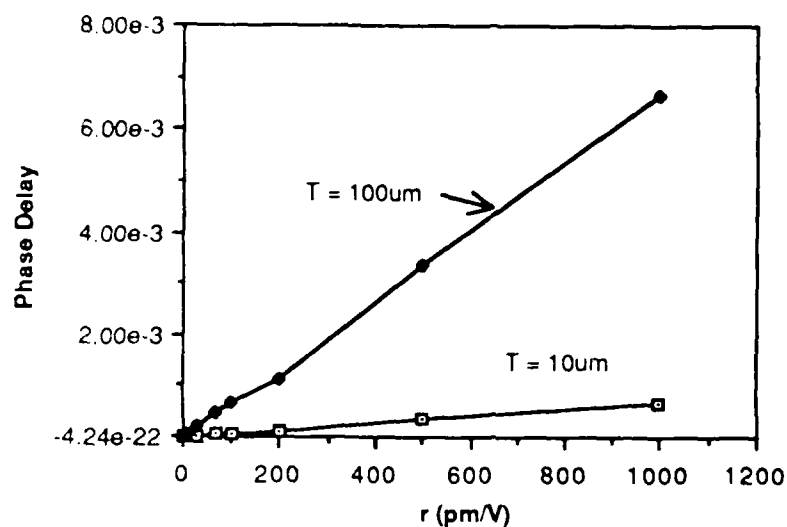
Figure 3a. Phase delay as a function of r, for various film thicknesses, field strength 10^3 V/m. The path length L was calculated assuming an incident beam angle of 20° from the normal.
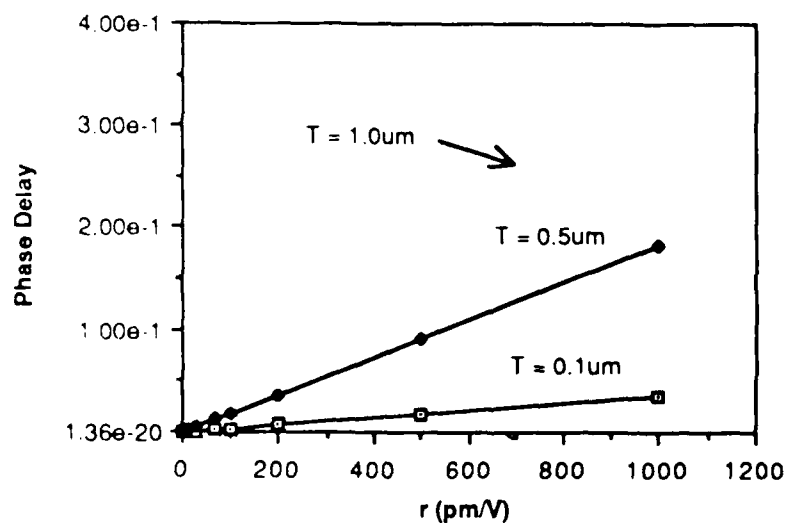

Figure 3b. Phase delay as a function of r, for various film thicknesses, field strength 10^6 V/m, dipole moment parallel to the surface. The path length L is twice the film thickness for a normally directed beam.

4.0 Conclusions

During the period of this contract, much progress has been made towards developing a highly ordered polymeric material which can be used for the electo-optic testing of integrated circuits. Although it was not possible to determine the linear electro-optic coefficients directly, the second harmonic generation experiments indicate that it should be possible to produce a film with an r_{33} coefficient which is at least an order of magnitude greater than the largest r coefficient for $LiNbO_3$. For an r_{33} of greater than 300 pm/V, with the dipole moments oriented perpendicular to the surface it may be possible to test integrated circuits using the vertical fringing fields, although the phase changes and resulting intensity changes will be on the order of one part in 1000 and an extremely sensitive optical detection system will be necessary to detect the modulation of the optical signal. If the dipoles are oriented parallel to the surface, an r_{33} of this magnitude will produce phase changes on the order of one part in 100, which will be easily detected.

Using the Langmuir-Blodgett deposition technique, methods are being developed to allow for deposition of materials with preferential alignment of the dipoles. By placing strong electric fields on the monlayer while it is on the subphase changes in surface pressure have been observed, indicating that ordering of the dipoles is taking place. By placing horizontal fields on the monolayer while it is on the subphase or during deposition on the surface of the device (using the existing conductors on the circuit) it should be possible to orient the dipoles parallel to the surface.

Using second harmonic generation as a primary diagnostic tool, work on the deposition of ordered dye films will continue. Once linear enhancement through a large number of layers (50-100) has been observed, the electro-optic coefficients of the films will be determined. Several other electro-optic materials are under consideration, especially electrochromic materials, which have become of much interest for use as display materials. [7,8]. Research on these materials will continue and once candidate dyes have been determined, they will be deposited using the L/B technique. Since the effect in these materials is one of

optical absorption and not phase modulation, changes in intensity are expected to be much larger than those seen in electric-birefringent materials. These materials may be suitable for the detection of weak vertical fields on the surface of integrated circuits, and will certainly be modulated strongly by the horizontal fields between conductors.

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